TIME DEPENDENCE OF THE HIGH TEMPERATURE CONDUCTIVITY OF SODIUM AND POTASSIUM β''-ALUMINA CERAMICS IN ALKALI METAL VAPOR

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ABSTRACT

'Inc. conductivities of sodium β''-alumina ceramic in low pressure sodium vapor and potassium β''-alumina ceramic in low pressure potassium vapor have been characterized in the four probe configuration by ac and dc measurements. Porous Rh/W films deposited by a photolytic process on Na β''-alumina and sputtered Mo films on K β''-alumina were used as electrodes. Alkali vapor pressures from 0.1 to 3.0 Pa and signal amplitudes generally less than 25 mV were used to maintain ohmic behavior over large ranges of temperature at the solid electrolyte sample during periodic measurements of conductivity vs. temperature. Conductivities dropped slightly for sodium β''-alumina ceramic and about 30% for potassium β''-alumina ceramic during the first 100 hours of test; the conductivities were then stable or rose slightly over 1550 hours at 1130 K and 600 hours at 1?(K) K for the Na and K materials, respectively. The high temperature conductivities vs. temperature were characterized for the equilibrated ceramics from below 900 K to above 1200 K; both ceramics showed good fits to the Nernst-Einstein equation over 400 K, with activation energies of 0.07 eV for sodium β''-alumina ceramic and 0.?-7 eV for potassium β''-alumina ceramic.

INTRODUCTION

High temperature applications of beta"-alumina solid electrolytes (BASE), including alkali metal thermal-to electric converters (AMTECs) require long term stability of BASE to liquid or high pressure vapor alkali metal on one side, and low pressure vapor of the alkali metal on the other. [1-5] This study addresses the issue of solid electrolyte stability to low pressure alkali metal vapor over periods of several hundreds of hours. The electrode materials used in this investigation have been characterized previously. [6-8] The stability of BASE to liquid alkali metal has been addressed in previous work from this and other laboratories, and the conductivities of Na-BASE and K-BASE in cells with two liquid alkali metal electrodes have been determined in intermediate temperature ranges. [9-12]

Many studies of the conductivity of sodium β"-alumina solid electrolyte (Na-BASE) ceramic have been carried out but few have characterized the conductivity above 800K; in addition, there has been no investigation of the time dependence of its conductivity at 10(K)-]?(K) K. [13-16] The generally accepted conductivity at high temperature was obtained by Weber using electrodes serving as Na^+ sources or sinks, and by Cole, Weber, and Hunt using proms Mo electrodes in a Na_g/argon atmosphere. [13-15] The latter measurements were obtained within one day of initial heating, with no

substantial change in the conductivity during this time.

Preparation of high quality potassium β''-alumina solid electrolyte (K-BASE) ceramic has had limited success, and only a few investigations of the conductivity of reasonably phase-pum ceramic have been reported.[11,17,18] Single crystal potassium β''-alumina shows high conductivity between 298K and 800K. [19-21] K-BASE ceramic is a good high temperature ionic conductor but is only moderately conductive near room temperature. [11,17] Its conductivity may be adversely affected by minor constituents such as potassium β-alumina, which is known to be a rather poor ionic conductor.[11, 17, 22-24]

The results of this work provide supporting evidence for the stability of the two stabilized alkali beta''-alumina phases at well defined alkali metal activity and temperature, although thermodynamic stability cannot be proved by a kinetic study. Potassium beta''-alumina with lower valence stabilizing ions such as Mg^{2+} or Li^{+} in the spine] block is almost certainly a thermodynamically stable phase at the temperatures at which a potassium AMTEC would be most useful (800" K to 1100 K). [11, 25] The upper temperature range of its stability is not known precisely, but is thought to be lower than that of spinel block stabilized sodium beta''-alumina, which is apparently stable at high Na_2O activity up to > 1900 K. [11, 13, 14, 16, 24, 26-28] There is, however, some disparity in

the results of studies of the stability of sodium beta"-alumina at high temperatures.[26,27]   Most studies of potassium aluminate. phases have investigated the simpler ternary K-Al-0 system, and little work on spinel block stabilized materials has been reported.[11, 29, .30, **31**, 32., **33**] Work in our laboratory has shown that the temperature regime at which stabilized potassium beta"-al[lmina becomes unstable with respect to loss of K_2O is about 1400 K in KCl vapor and about 1900 K at high K_2O and O_2 activities. This paper provides evidence for the prolonged kinetic stability of stabilized potassium beta''-al umina at 1200 K, and supports a hypothesis of the thermodynamic stability of both sodium and potassium beta''-alumina at pressures of O. 1 to 3.0 Pa alkali metal vapor at 1150 and 1200 K respectivel y.

EXPERIMENTAl.

Cylindrical Li$^+$-stabilized Na-BASE tube.s nominally 15 mm in diameter with a 1 mm wall thickness were obtained from Cerametec, Inc, Salt l ake City, UT. Similar Na-BASE tubes 15 mm or 25 mm in diameter, were exchanged with KCl vapor at 1425 K in vacuum or at 1625 K with KCl vapor in a K-BASE powder bed in air at 1 atm. The two K-BASE exchange processes produced somewhat dissimilar ceramics and these will be ident i fied as K-HAS};(1425) and K-BASE(1625).

Four thin film metal electrodes, shaped as either rings or patches with areas from about $4 mm^2$ to about $5 cm^2$, were deposited cm Na- and K-BASE tube sections. Rh$_3$W electrodes were deposited on Na-BASE using a photolytic chemical vapor deposition process. Sputter- deposited Mo fil Ins were used as electrodes on K-BASE.] electrodes were contacted with Mo or Ni meshes tied on with Mo leads and were separated by regions of uncoated BASE. Details of the W/Rh photolytic deposition process, the. K- BASE exchange process, and AMTEC electrode studies are presented else.whe.j c. [6-8, 25, 34]

Sodium and potassium were obtained as regent grade metals under argon, and were purified by heating the liquid metal with pieces of Na- BASE and K-BASE respectively, and filtering the liquid metal through sequential $7 \mu m$ and $2 \mu m$ stainless steel filters. This technique removes particulates and lowers the contamination level of the alkali metal by Ca, which displaces the alkali and reduces the ceramic conductivity y. The vapor pressure of the alkali metal was calculated using establ i shed expressions for the sat urated vapor pressure above the liquid at the cold end and correcting for atomic velocity change due to the temperature difference bet wcen the pool region and the elect rol ytc region. [35-38]

The test apparatus, shown in Fig. 1 was a stainless steel tube with feedthroughs for leads, thermocouples, pump out, argon backfill, and introduction of a

controlled quantit y of alkali metal. Samples were mounted on a central support tube with α-alumina standoffs over a Mo foil wrap. 1 lectrical leads and shielded type K thermocouples were insulated with a-alumina tubing inside the test apparatus. Finall y, refractory metal foils (Zr, **Mo,** 'l'a) were inserted inside the stainless steel t ube for gettering and protection of the samples from high vapor pressure components of the steel. The entire assembl y was welded shut, The small gaps between the outer steel feedthroughs and a-alumina insulators were hermetically sealed with a slow setting epoxy (Iipvxi-Pate.h"), The apparatus was set up with a pool of alkali metal at one end of the tube. assembl y. The temperature of the pool was controlled with heating tapes to temperatures up to 600 **K,** and the temperature measured with a thermocouple in contact with the pool. The assembly was placed in a tube furnace with the alkali metal end projecting out of the furnace; electrolyte temperatures up to 1400 **K** with only small gradients across the samples could be achieved.

Conductivities of the BASE samples were measured in a four probe. configuration by ac and dc techniques. Conductivity was measured using two equipment set-ups. in one., a Solartron 1286 and 12S5 frequency anal yzer and electrochemical interface with a digital voltmeter controlled by a IBM **X'1'** computer were used to measure the conductivity by elect rochemical i mpedance spcct roscopy, in the other, a **PAR** 173 potentiostat/galvanostat was used in galvanostatic mode, with a PAR 175 universal programmer and a PAR 176

current follow. In the latter case, a slow triangular current signal was imposed between the outer pair of electrodes with the 176 output monitored by on the x channel of an Hewlett-Packard 7047A x-y recorder and the potential between the inner pair of electrodes directly monitored by the y channel. Alkali vapor pressures from 0.1 to 3.0 Pa and signal amplitudes generally less than 25 mV were used in the reported measurements; ohmic behavior was maintained over large ranges of temperature at the solid electrolyte.

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Electrolyte samples were held at the specified temperature but conductivity and other measurements were made periodically as the temperature was cycled. Computed exposure time excludes time at temperatures more than SO K lower than the reported exposure temperature. "1'here were several periods of stand at much lower temperatures (775 - 875 K); conductivity was unaffected by cooling and reheating.

After the samples were held at high temperature for 15S0 hours (Na-BASE) and 600 hours (K-BASE), the low temperature end of test cell was cooled to room temperature for several hours to drive alkali metal inclusions/defects from the electrolyte samples; the test apparatus was then cooled to room temperature, filled with argon, and cut open. X-ray diffraction measurements on K-BASE were performed before and after the test.

RESULTS AND DISCUSSION

Measured conductivities of Na-BASE and K-BASE were analyzed with respect to electrolyte temperature, ambient alkali metal pressure, and elapsed time at the high temperature. Conductivities were measured over a large range of ambient alkali metal pressures. It was found that very low pressures (<< 0.1 Pa) gave rise to non-ohmic responses. Furthermore, high potassium pool temperatures resulted in reduced conductivities as impurity sodium pressures increased; likewise low sodium pool temperature es resulted in high potassium impurity pressures and reduced measured sample conductivities. The latter effect begins below 0.5 Pa sodium pressure, but the effect is significant only below 0.3 Pa sodium pressure.

The Conductivity of Na-BASE

'The conductivity of Na-BASE was unaffected by ambient sodium vapor pressures between 0.3 and 2.0 Pa; data taken over this pressure range have been used in the determination of the temperature and time dependence of Na-BASE conductivity, shown in Figs. 2 and 3, The temperature dependence of Na-BASE conductivity from 1000 K to 1200 K, also shown in Fig. 2, was found to vary systematical y over only about 5%, and conduct ivities measured at temperatures from 1028 K to 1201 K were used to evaluate the time dependence of Na-BASE

conductivity. There was no apparent change in the activation] energy of Na-BASE conductivity with time. The high temperature activation energy of Na-BASE, 0.067 eV, was determined from the plot in Fig. 2. It is significantly smaller than the value of 0,115 eV reported in 1979 by Cole, Weber, and Hunt on Cerametec Na-BASE, although the magnitudes of the high temperature conductivity arc similar. [15] Cerametec has changed its processing procedure in the intervening time; this change may be responsible for this difference in Na-BASE ceramic conductivity.

The data taken early in the test, shown in the inset of Fig. 3, were measured galvanostatically and indicate that the them is a small decrease in the magnitude of the conductivity in the first hundred hours at high temperature. Data points from later in the test were generally determined from the low frequency limit of the impedance response, and displayed greater scatter and no systematic time dependence over 1450 hours. Previous X-ray diffraction (XRD) analysis of Na-BASE has indicated that no significant degradation of the sodium @"-alumina occurs following cycling from 300 K to 1273 K and back in vacuum. [10, 39, 40] Several very weak peaks appeared in the Na-BASE diffraction pattern following XRD measurements at 1273 K in vacuum for about 5 hours, but they were not β-alumina peaks, and could not be reliably assigned. [40, **41**] The Na-BASE appeared free of p-alumina by XRD both before and after cycling to high temperature. [4 0]

Higher potassium pool temperatures resulted in systematically higher measured elect rol yte conductivities, but there was little ten iperature dependence bet ween 0.1 and 3.0 Pa. The conductivities of K-BASE(1425) and K-BASE(1625) electrolytes were measured with respect to temperature at nearly constant ambient potassium pressure on initial heat-up, after 50, 120, 300 and 500 hours. No 300 hour data point for the 1425 K electrol yte was included because a lead had shorted to ground during the measurement. The temperat urc dependence at each time was fit to the Nernst-Einstein equation. [42] The conductivity at 1000 K was calculated from the fit to the data and plotted against time, as shown in **Fig.** 4. The. conductivity of both samples decreased over the first 100 hours and then slowly rose with time. The activation energy for conductivity y after the initial 100 hours was determined to be 0.267-0.28 eV for both samples, as shown in **Fig.** 2. The high temperature activation energy for K-BASE(1425) increased with time for the three samples measui ed, while the activation energy for the K-BASE(1 *625)* decreased slight] y. The conductivity at T> 900 K was always higher for the 1625 K material than for the 1425 K material. The K-BASE(1425) activation energy deereases during the same time period that the overall conductivity drops and shows little change from *120* to *500* hours.

The majority phase in both 1425 K and 1625 K ceramics remains β "-alumina, indicated both by the high conductivity y and the XRD pattern. XRD of K-

BASE(1625) ceramic surfaces before and after the test showed that there was a increase in the minority p-alumina phase from 1 -2% to 3-4 %. The limited formation of the beta phase almost certainly occurred transiently and coincidentally with the initial conductivity drop, and in limited regions of the ceramic, since its continuous formation in the bulk ceramic would have resulted in a continuous drop in conductivity of the K-BASE. Because formation of the beta phase as a thin surface phase would have little effect on the four-probe conductivity measurement, we believe that the beta phase is formed at the grain boundaries, possibly via K_2O loss from $KAlO_2$ initially present at the grain boundaries, and concomitant void formation. The slow increase in conductivity may be due to voids collapsing at the grain boundaries. Information of a very thin surface film is not ruled out, but because XRD samples the surface preferentially, a thick layer of the beta phase would have dominated the XRD pattern. It is fairly well established that commercial Na-BASE has NaAlO2 at the grain boundaries, and the aluminate phase is refractory enough to persist through the ion exchange process. [43]

'The activation energy is similar to that of single crystal potassium p-alumina (0.28 eV), although the K-BASE conductivity is several times higher than that of single crystal $K-\beta$-alumina and possibly 20-50 times higher than the conductivity of K-p-alumina ceramic extrapolated to similar temperatures. [11, 14, 16, 23]

The pm-exponential and activation energies used to fit the high temperature conductivities of the Na-BASE and K-BASE electrolytes are given in **Table 1.**

The decline in conductivity at the beginning of the tests was fit to an exponential function. The time constant $(\tau_{1/e})$ for Na-BASE is 44 hours, using on] y the galvanostaticall y controlled dc measurements from the first 100 hours of test. The time constant for K-BASE is 37.5 hours, using the values of the conductivity at 1000 K calculated from the fits of the conductivity/temperature data to the Nernst-Einstein equation. Conductivity determined for Na-BASE from impedance measurements is not included in the calculation of conductivity decline because there were few impedance measurements made in the first 100 hours and because there is substantially less scatter in the galvanostatic data. The later ac measurements of the conductivity of Na-BASE showed higher scatter and no significant trend with time, although the average value of the conductivity was higher than the final value at 100 hours given by the fit to the early dc measurements.

CONCLUSIONS

The activation energy of Li^+-stabilized Na-BASE at high temperature is similar to the activation energy of sodium β''-alumina single. crystals at higher temperatures and is generally consistent with earlier results on Na-BASE ceramic

to 1273 K. [15] We find conductivities of similar magnitudes but a significantly lower activation energy at high temperature. It is possible that the difference is due to a minor change in the sodium p-alumina ceramic provided by Ceramatec Na-BASE since 1979, A small decline in conductivity early in the test may be due to loss of some Na_2O, with the conductivity later in the test either stable or improving at a very slow rate. The conductivity of Na-BASE is very stable after a small initial decline, and there is no reason to expect that conductivity decline will limit long term AMTEC operation, At low AMTEC condenser temperatures, some reduction of the Na-BASE conductivity may occur if the sodium working fluid is not free from potassium contamination and the ambient sodium pressure in the cold zone is less than about 0.3 Pa.

The conductivity of Li^+-stabilized K-BASE at high temperatures (600-1200 K), after 100 hours at 1200 K, has an activation energy very close to that observed for the conductivity of single crystal potassium p-alumina from 298 K to about 700 K. However, the K-BASE conductivity is several times higher than that of single crystal potassium p-alumina extrapolated to high temperature and much higher than would be expected for polycrystalline potassium p-alumina ceramic. The grain boundaries of K-BASE may be expected to contain K/Al/O phases without Li+, and long exposure at test conditions probably results in K_2O loss and β-alumina formation at the grain boundaries in the absence of a @"-alumina stabilizing ion. The ionic conductivity of K-BASE equilibrated with potassium vapor at high temperatures is high enough for its use in efficient potassium

AMTEC devices from *900* K to 1200 K. 1 n the lower part of this temperature range, it is important that the potassium working fluid be as free from sodium as possible.

ACKNOWLEDGMENTS

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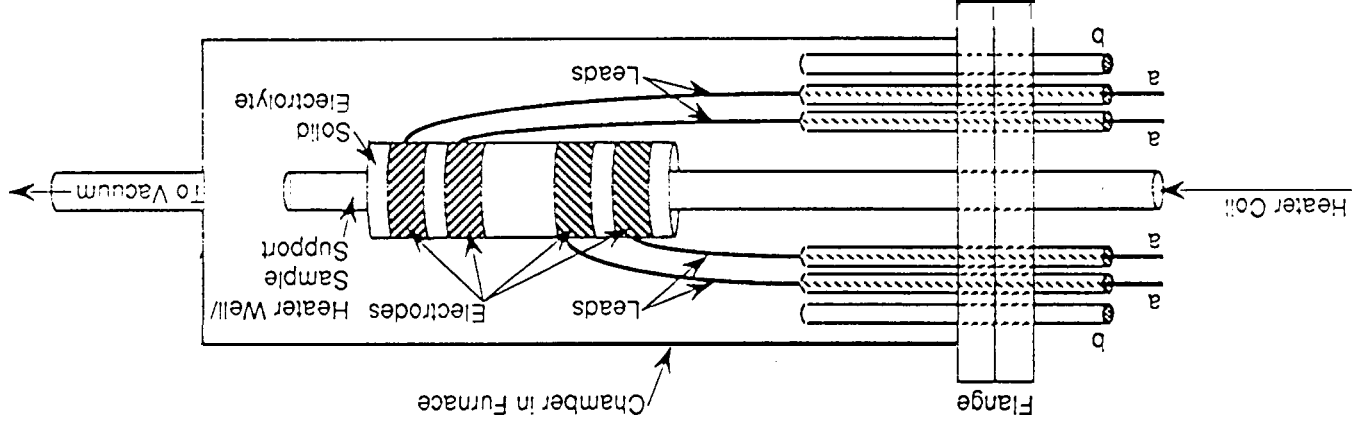
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TABLE 1. Pre-exponentials and activation energies from Nernst-Einstein fit to high temperature conductivity of Na-BASE and K-BASE

Electrolyte	$Sigma_0$	E_{act}	E_{act}
	K/(ohm-cm)	kJ/(mol-K)	eV
Na-BASE			
t=0hrs [21]	1.98×10^3	11.9	.115
t=100-1550 hrs	$1.27(19)x10^3$	6.50(13)	.067(12)
K-BASE			
1425 K #1,2			
t=O hrs [98]	$2.89(29)x1\ 0^3$	18.0(8)	.1 86(8)
142S K #2			
t=100 hrs	$3.60(13)x103$	27.0(3)	.280(3)
142S K #3			
t=O hrs	$2.71(77)x1(?$	23.7(2.7)	.246(28)
t =600 hrs	$3.9(1\ .2)X10^3$	25.8(2.3)	.267(24)
1625 K			
t=O hrs	$1LO(2.0)X10^3$	29.1(1.6)	.302(16)
t= 120-600 hrs	$5 .06(34)x\ 10^3$	26.0(6)	.269(6)

FIGURES

Fig. 1,   Schematic diagram of the alkali metal vapor exposure test chamber.

Fig. 2,   Temperature dependence of the conductivity of Na-BASE, K-BASE 1425, and K-BASE 1625, after >100 hours of exposure to alkali metal vapor at high temperature.

Fig. 3,   Time dependence of the conductivity of Na-BASE, 1028-1201 K, at 0.3 to 2.0 Pa Na_g. The inset shows the initial 100 hours on an expanded scale.

Fig. 4,   Time dependence of the conductivity of K-BASE at 1000 K, calculated from fits to conductivity vs. temperature data, at 0.1 to 2.0 Pa K_g.

FIG 2

filename : kNaSGv16.spg

FIG 3

CONDUCTIVITY (ohm-cm)⁻¹

0.213 $(\Omega-cm)^{-1}$

0 153 $(\Omega\ cm)^{-1}$

TIME (hours)
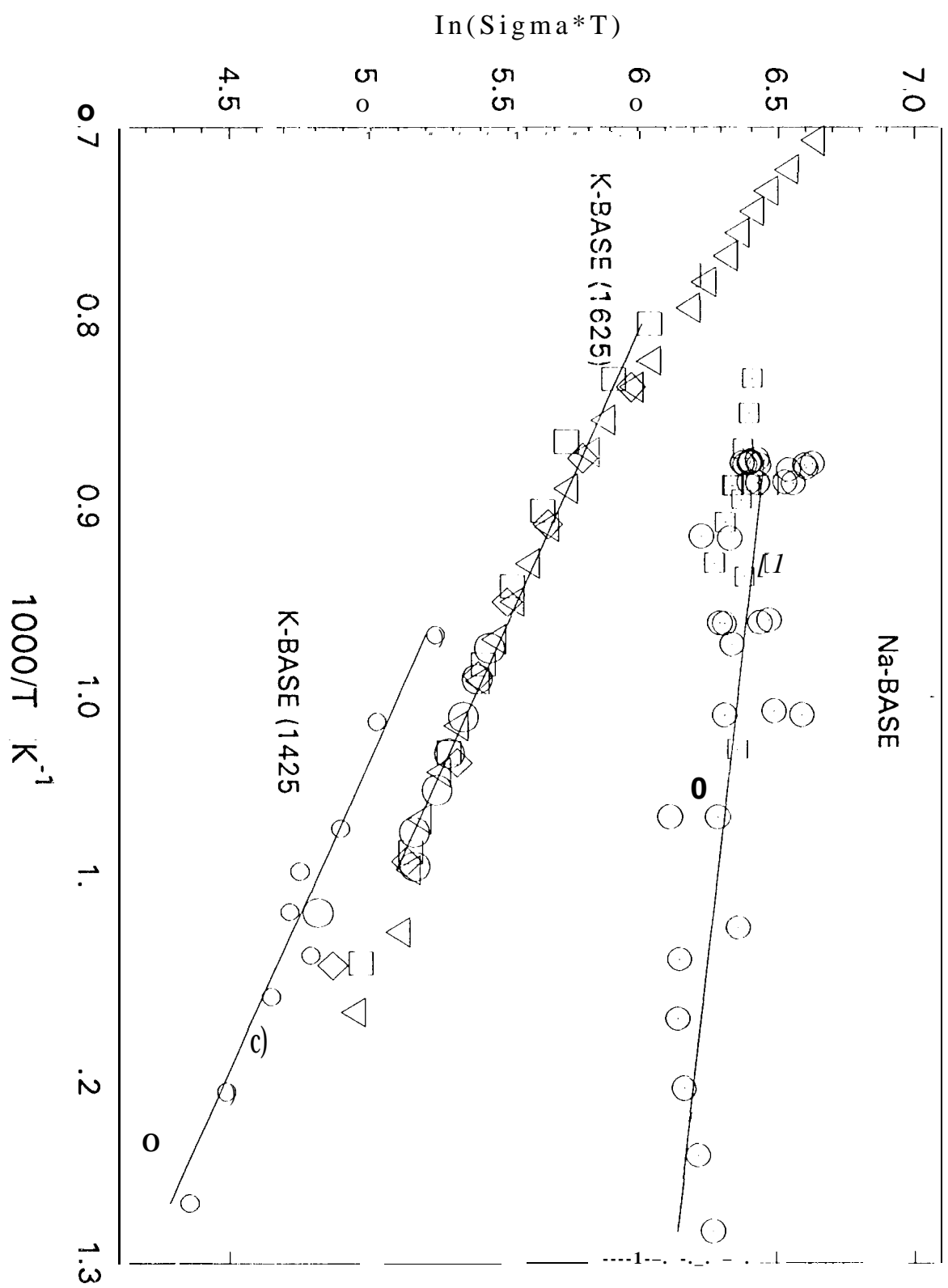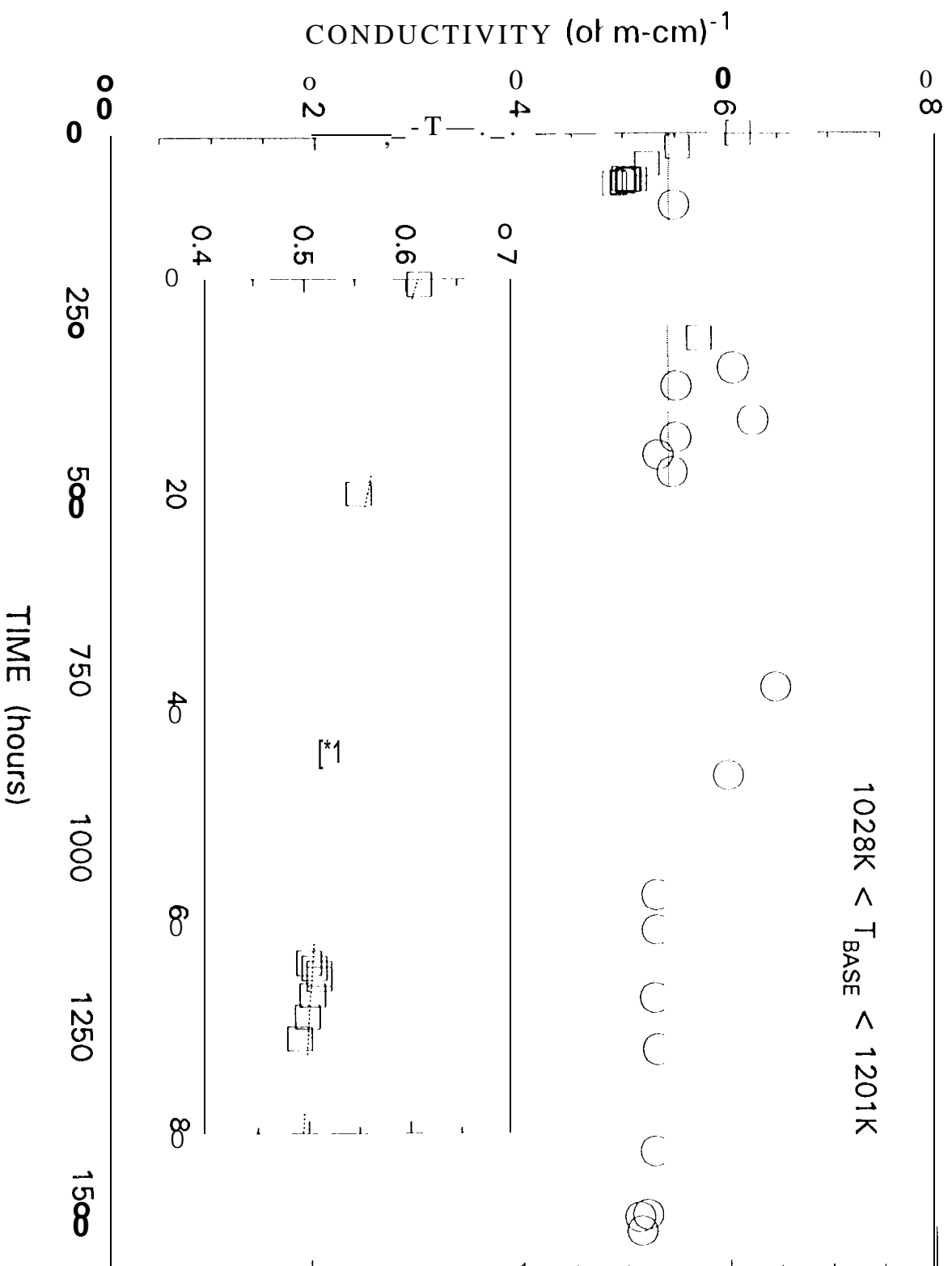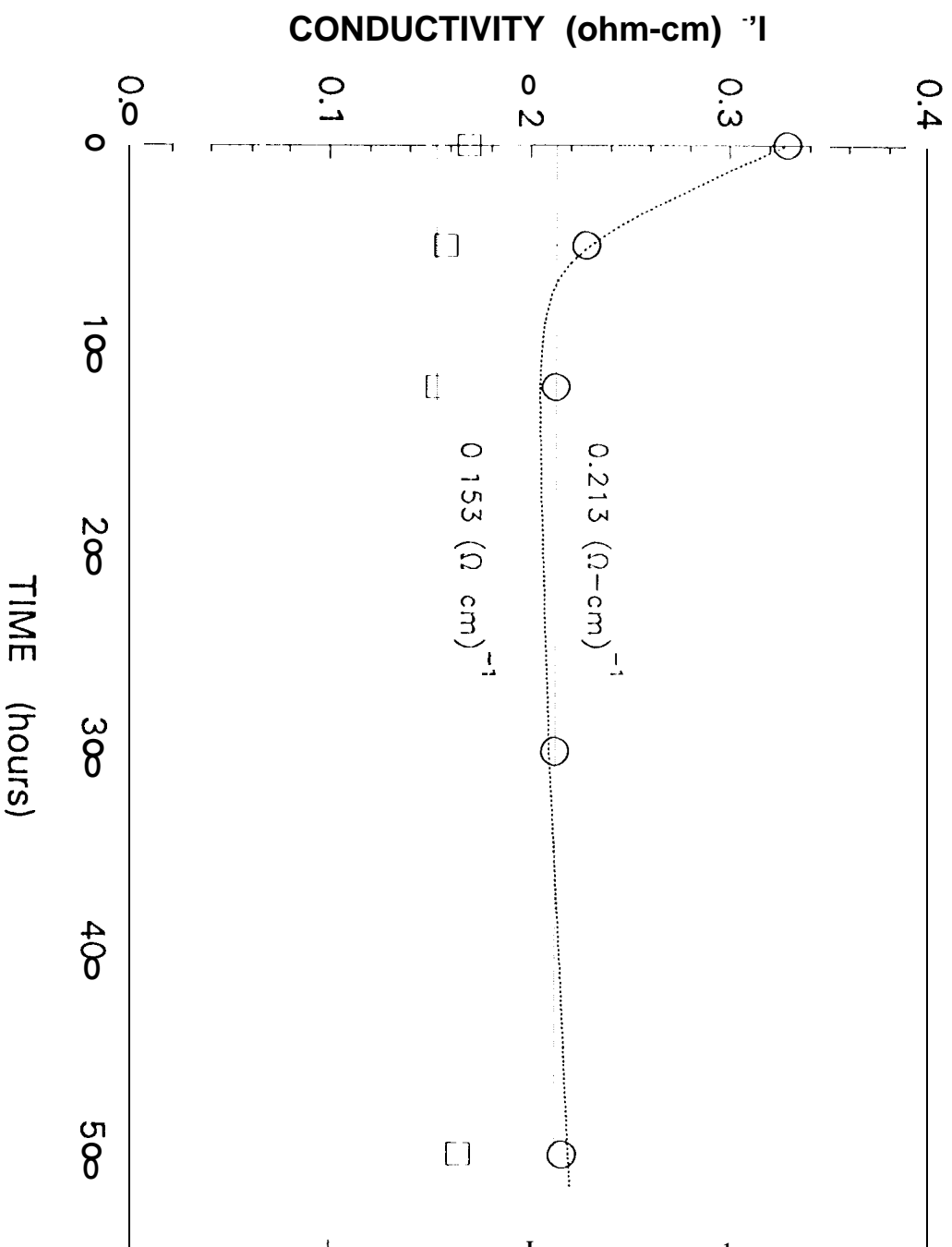
Ionic conductivity (t, 1000K) taken from fits to Sig(T) = $(Sig_0/T)exp[-E_{act}/k_bT]$ for Cond(T) data taken at 0, 50, 120, 300, and 500 hours of exposure of K-BASE to about 1.0 Pa K_{gas} at 1200K.

FIG 4